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U.S. Environmental Protection Agency
CLP Sample Management Office
P.O. Box 818, Alexandria, Virginia 22313
PHONE: (703)/557-2490 or FTS/557-2490

SAS Number
7084 E

SPECIAL ANALYTICAL SERVICES
Client Request

☐ Regional Transmittal ☐ Telephone Request

A. EPA Region/Client: Region V/ARCS, E & E
B. RSCC Representative: Jan Pels
C. Telephone Number: (312)353-2720
D. Date of Request: 2/7/92
E. Site Name: NL Industries, TaraCorp Lead Smelt Site, Granite City, IL K7
ATSDR Multistate Lead Exposure Study

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

Analysis of cadmium and lead by ICP emission spectroscopy of bulk dust from private residences. Results must be reported as mg/kg cadmium or lead on an as received basis after removal of extraneous material. Total solids is not to be determined for these samples. Weigh and record the total weight of each sample received, in grams to the nearest 10 mg. If the sample size is inadequate after initial preparation (<1.5 g), the alternate graphite furnace atomic absorption (GFAA) methodology must be used in order to utilize small sample aliquots (<500 mg)*. For those samples containing suspected paint chips or peeling paint particles, the paint chips must be separated, weighed, and analyzed for Cd and Pb. Cd and Pb results must be reported in mg/kg for each sample portion and for a mathematical composite value for the total dust sample. Extraneous material will also be separated from the sample and weighed. Document the nature and character of each portion of the sample. Each portion of the sample including dust, extraneous material and suspected paint chips must be described and documented in the data package. The description of the dust portion must include an estimate of the amount of dust and fibrous material (lint) in the sample after the sample has been ground. The descriptions must also include any unusual characteristics that will assist in the evaluation of the final results. An example of a completed form 1 including sample descriptions is included as an attachment to this SAS. The laboratory must also include a detailed case narrative in the deliverable package. This case narrative must include a description of any problems encountered during sample preparation and during sample analysis. Any unusual sample characteristics must also be included to assist in the Regional validation of the sample package. An example of a case narrative is included as an attachment to this SAS.

*See attached GFAA SAS

2. Definition and number of work units involved: 355 dust samples

The investigative samples and field duplicates will be bulk dust from residential homes. Sample sizes will vary between 0.2 g and 20 g. Most will be in excess of 3 g. Only 10%-15% of the samples are expected to be less than 1.5 g. To improve precision of analysis, the samples will be homogenized using a SPEX 8000 Mixer/Mill or equivalent. The laboratories are to be cautioned that each sample has the potential to be subdivided into three portions. This is a time consuming procedure that can double the number of samples to be assayed. SMO and Region may initiate a conference call with the selected laboratory to assure that all parties understand the scope and expectations for this project. SMO and Region 5 may also visit the laboratory during the initial analyses to audit SAS analytical specifications, to provide corrective actions, and to minimize problems associated with subsequent data reviews.

3. Purpose of analysis:

Superfund ATSDR Multi-State Lead Exposure Study

4. Estimated date(s) of collection: September 4 - October 4, 1991

5. Estimated date(s) and method of shipment: Federal Express - upon award of SAS contract

6. Number of days analysis and data required after laboratory receipt of samples: **Laboratory data rejection and nonpayment will be recommended if the methods outlined in this SAS are not followed by the laboratory.**

Data due 35 days from receipt of samples.

7. Analytical protocol required:

The samples are expected to be dry, bulk dust. Record the weight of the total dust sample as received to the nearest 10 mg. A low power stereomicroscope will be used to inspect the sample for paint chips and other materials. A detailed sample description will be included in the raw data and on Form 1 for each sample portion. Extraneous material such as carpet fibers, tobacco products, jewelry parts, small metal parts, coins, and crayons must be removed from the sample (use clean or Teflon coated forceps), weighed, and archived. It is important that metal parts are removed from the samples. (Crayons will form a wax and dust ball during grinding and will cause a nonhomogeneous sample.) Some of the samples will contain large amounts of hair and carpet fibers. In such cases it may not be possible or desirable to separate the fibrous materials as extraneous material. The removal of too much fibrous material may cause the removal of most of the dust. The grinding process will tend to separate the dust and fibrous materials. Paint chips and suspected paint chips must also be separated from the sample and weighed. The use of a low power microscope is mandatory to determine if small chips are in fact paint, plastic, or plaster. If the suspected paint chips are obviously paint, these shall be noted as probable paint chips on the Form 1. After the various materials have been separated from the samples, the weight of dust, extraneous material, and paint chips must be recorded on forms in the raw data. The dust sample portion and suspected paint chips must be homogenized using a SPEX 8000 Mixer/Mill or equivalent. After the dust portion has been ground, it may contain "lint balls" and dust. Only the dust will be taken for digestion. Dust samples containing less than 1.5 g must be

assayed using the GFAA SAS. If the weight of paint chips is less than 100 mg, the sample is not to be homogenized since the entire sample may cling to the mill and will not be recoverable. Paint chip samples with weights less than 100 mg must be digested without prior grinding. The entire sample may need to be digested. Very small samples of paint chips (5 to 50 mg) shall be prepared and analyzed using the GFAA SAS. The samples must be digested using SOW 7/88 or ILM01. All dust samples remaining after homogenization and digestion will be archived until Regional review of the data is complete. Digestion logs should also indicate when a sample is not homogenized prior to digestion.

8. Special technical instruction:

The laboratory will provide the following information with the bid package to SMO for each ICP instrument to be used: 1) instrument manufacturer and model, 2) instrument detection limits for each Pb and Cd line that will be used, 3) linear ranges for all Pb and Cd lines to be used, and 4) interelement correction factors and background correction points for all Pb and Cd wavelengths selected for use. The laboratory must address IECs for Cu, Fe, Cr, Al, [redacted], V, Ti and Mo for Pb and Al, Fe and As for Cd. All of this information must be submitted with the bid package and will be reviewed prior to selection of a laboratory.

9. Analytical results required: original tags, airbills, etc. must also be submitted,

Data deliverables will be in accordance with ILM01 including notations for any samples requiring separation, homogenization, separate and composite values for dust, paint chips and total sample values. A floppy disk deliverable is not required. The Form 1 will also describe the weight, character of each sample received as well as noting any problems due to extraneous materials.

10. Other

Examples of Form 1, sample weight logs, and sample description logs are included as attachments. Their use is mandatory. The information included on these forms must be included in the case by the laboratory, along with all other SOW required forms and deliverables.

11. Name of sampling/shipping contact: Cathy Kouris, E & E
Phone: (312)663-9415

12. Data Requirements

Parameter:	Detection Limit	Precision Required (±% or conc.)
Cd	5 ug/l in the digest or 1.0 mg/kg (as received)	30% RPD for duplicate samples greater than 10 mg/kg or a duplicate difference <2 mg/kg for results less than 10 mg/kg
Pb	2 X ICP IDL (ug/l) in digestate, which must be less than 100 ug/l.	30% RPD for duplicate samples greater than 700 mg/kg, or a duplicate difference <200 mg/kg for results less than 700 mg/kg. 50% RPD for duplicate samples greater than 1000 mg/kg

II. QC Requirements – Per the SOW, with the following modifications:

Audits Required	Frequency of Audit	Limits* (% or conc)
Initial + cont. Calibration Blanks	As per SOW 7/88 or ILM01	<5 ug/l Cd <100 ug/l Pb (ICP)
Initial + Cont. Calibration Verification	As per SOW 7/88 or	90% to 110% recovery
Lab Duplicates	1 in 10 samples or one per digestion set whichever gives the greater number of duplicates	See item 12. If limits are exceeded, rerun the sample and duplicate. If the limits are still exceeded, call SMO for guidance.
Matrix Spike* (Digested) 100 mg/kg Cd 500 mg/kg Pb	same as lab duplicate	75% to 125% recovery. If the limits are exceeded, rerun the sample and spike. If the limits are still exceeded, contact SMO.
Prep Blank	1 in 20 samples or one per digestion set which ever gives the greater number of blanks.	<5 ug/l Cd and <100 ug/l Pb If blank limits are exceeded, redigest those samples with Pb or Cd concentrations <1000 ug/l Pb or <50 ug/l Cd.
Solid LCS	same as prep blank	As per SOW if the CLP solid LCS is not available, an independent liquid LCS is acceptable. This must be documented in the case narrative.
Serial Dilution CRDL Standard ICSA and ICSAB	As per SOW	As per SOW

*If the matrix spike concentration is less than 50% of the sample concentration, the 75% to 125% recovery is not mandatory.

It is intended and required that the resulting data will not be qualified by the out of control sample duplicates and matrix spikes. With careful sample preparation, homogenization, and aliquot selection, the above QC criteria should be met with minimal reanalyses and redigestions.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

If QC limits are exceeded after corrective action or redigestion, contact SMO and Region 5

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for Special Analytical Services. Should you have any questions or need any assistance, please call the Sample Management Office.

IV. Attachments:

The following three forms are required as part of the data reporting package. All of the information included on these forms must be included in the data package from the laboratory. Examples of completed forms are also attached, along with an example of a case narrative. The GFAA SAS is also attached.

U.S. EPA - CLP

1

EPA Sample No.

Lab Name:_____ Contract:_____

Lab Code:_____ Case No:_____ SAS No:_____ SDG No:_____

Date Recieved:_____ Lab Sample ID:_____

Total Sample Weight (g):_____

Weight of Dust and Fibrous Mtl (g):_____

Weight of Extraneous Material (g):_____

Weight of Suspected Paint Chips (g):_____

Concentration Units: mg/kg

	Cadmium	C	Q	Lead	C	Q
Dust						
Paint Chips*						
Composite Result						

Sample Description:_____

Comments:_____

* Paint Chips (check one):

Suspected ☐ Probable ☐

Sample I.D.	Total Weight (g)	Paint Chip Weight	Weight of Dust (g)	Comments

Net Sample Weights Worksheet

Analyst:_____ Date:_____

Balance Checked:_____

Sample I.D.	Chip Tare Weight (g)	Total Chip Weight (g)	Net Weight of Chips (g)	Tare Weight Ext Mtl (g)	Total Weight Ext Mtl (g)	Net Weight Ext Mtl (g)

Extraneous Material (Ext Mtl) and Chip net weights are also recorded on the sample description worksheet

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1

EPA Sample No.

Lab Name: EXAMPLE Contract:

Lab Code: Case No: SAS No: SDG No:

Date Recieved: Lab Sample ID:

Total Sample Weight (g): 4.49

Weight of Dust and Fibrous Mtl (g): 4.43

Weight of Extraneous Material (g): 0.02

Weight of Suspected Paint Chips (g): 0.040

Concentration Units: mg/kg

	Cadmium	C	Q	Lead	C	Q
Dust	36	P		5800	P	
Paint Chips*	170	F		18000	F	
Composite Result	37			5900		

Sample Description: An examination of the total sample revealed dust, carpet fibers and blue and white suspected pait chips. The suspected paint chips were separated. Plastic and plant parts were removed as extraneous materials. The remaining dust portion was ground and yielded a sample that was 50% dust and 50% lint.

Comments: After consulting Region 5, the Cd and Pb results for the dust sample were reported as an average of three determinations.

* Paint Chips (check one):

Suspected ☐ Probable ☒ X

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ICP-10

— EXAMPLE —

U.S. EPA - CLP

1

EPA Sample No.

Lab Name: EXAMPLE_____ Contract:_____

Lab Code:_____ Case No:_____ SAS No:_____ SDG No:_____

Date Recieved:_____ Lab Sample ID:_____

Total Sample Weight (g): 27.88_____

Weight of Dust and Fibrous Mtl (g): 25.59_____

Weight of Extraneous Material (g): 1.74_____

Weight of Suspected Paint Chips (g): 0.55_____

Concentration Units: mg/kg

	Cadmium	C	Q	Lead	C	Q
Dust	8.9	P		360	P	
Paint Chips*	15	P		2900	P	
Composite Result	8.9	-		370	-	

Sample Description: The examination of the total sample revealed dust, carpet fibers and suspected paint chips. Paper, wood and plant materials were removed as extraneous materials. Some blue suspected paint chips were separated. The dust portion was ground and yielded a sample that was 90% lint and 10% dust._____

Comments:_____

* Paint Chips (check one):

Suspected ☐ Probable ☒ X

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— EXAMPLE —

CASE NARRATIVE
NL INDUSTRIES
ICAP for Pb and Cd in dust
SF0819

Fifty nine dust samples were submitted for analysis by ICAP for Pb and also Cd.

Pre analysis preparation

Upon receipt the samples were examined under a low power stereomicroscope. Notations were made as to contents of the clear plastic bags containing the dust samples. For each sample, a total sample weight was determined and recorded. Next the sample was physically separated into fractions - a dust fraction, a suspected paint chip fraction, and an extraneous material fraction. Each fraction was weighed and the weights recorded. The dust fraction and the suspected paint chip fraction were each separately ball milled using plastic balls and containers. The extraneous materials were not milled or analyzed. Approximately half gram aliquots were taken for the dust fraction digestion. If possible, twenty to fifty milligrams of ground suspected paint chips were used for digestion. Some of the suspected paint chip samples contained less than 20 milligrams. In these cases, the entire sample was digested. The CRL SOP for soil samples using microwave digestion with nitric acid and yttrium added as an internal standard was used to prep these sample fractions. A description of the milled dust portion of each sample was included on the Form 1 report. The relative volumes of lint and dust as percentages were included in the descriptions. "Lint" is used to describe any fibrous material remaining after milling.

ICAP Analysis

Method development: Based on the analysis of samples from another site and from initial attempts at analyzing SF0819 samples, it was found that there appeared to be a stray light interference from Cu affecting the Pb 220 nm line when using the TJA 1160 ICAP unit. Cu was found at concentrations high enough to affect Pb results in a number of dust samples. A decision then was made with Dr. J. Morris to use the TJA ICAP 61 unit where this kind of interference was not observed (see attached scans illustrating this).

Based on the previous work mentioned above, an instrument run method was created for the TJA 1160 to determine Pb and Cd in digests containing yttrium as an internal standard. All the element lines selected for this method are used in the standard CRL SED5 or SED5Y run methods. The lines selected essentially used the same limits, background correction points, and calibration standards that were used for SED5Y. Other elements selected included Ca, Mg, and Na, which were to be viewed as indicators of "high solids" content; As, Al, Fe, Cu, Ti, and Mo were selected for purposes of IEC corrections.

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ICP-12

A similar run method, DUSTY61, based on this 1160 run method was created and used with the TJA ICAP 61 but additional element lines and different background correction points were selected. Selection of these points was based on previously run element scans and noted observations of these scans. For both Pb and Cd an additional line was available and selected as a "back up" line (standardized, monitored, but not reported). Pb values were reported using the 220 nm line and for Cd the 228 nm line was used. The Pb backup line was the 182 nm line and for Cd the backup was the 214 nm line. A copy of the method is included with the Case deliverables. Nitrogen gas was used to purge the torch compartment optics (a flow setting of approximately 10 psi was used).

ICAP Analyses: The actual analysis run numbers for the ICAP 61 unit were Runs 952A, 954A, 957A, 958A, 960A, 963A, 965A, 966A, and 976A. Each run was conducted on a different day. The earlier runs included prepared dust samples while the later runs included the prepared suspected paint chip samples.

Reporting of results: Since the work performed for this project can essentially be considered more experimental than routine in nature, the standard Case deliverable protocols were not entirely followed. A number of worksheet and reporting forms were designed by ESAT personnel and used for recording fraction weights, sample observations, and reporting of sample results for Pb and Cd.

Where appropriate, a composite value was reported for each sample for Pb and for Cd. The composite value was calculated by combining the dust fraction concentration and the suspected paint chip fraction concentration.

Per discussion with D. Payne and Dr. J. Morris, additional data reports were to be provided for the data user. These reports included results for digestion blanks, laboratory duplicates, matrix spikes, and laboratory control samples (LCS). For these latter reports, copies of the CRL **ENABLE** report forms designed by M. Jupp were utilized.

Comments: During the course of preparation of some of the suspected paint chips, the plastic grinding balls were inadvertently included. These prepared sample fractions were analyzed using GFAA; this was so noted on the report forms for the affected samples.

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Pb results for the NBS reference paint sample show low extraction (digestion) efficiency (50% and 73% Pb recovery). However, even with this lower extraction value the overall effect on the composite value was minimal to insignificant for the samples analyzed since the amount of paint chip material was small compared to the dust sample fraction. It should also be pointed out that throughout all the runs conducted, the LCS results were consistently (with one exception) within the control ranges specified by EMSL documentation for the LCS used. Even though the EMSL values may not necessarily reflect "true" values they do represent a comparative indicator of extraction efficiency.

Spex stock standards (1000 ug/ml and 10,000 ug/ml) were used to prepare working standards. Custom made solutions from Inorganic Ventures were used to prepare working check solutions. For the first seven analysis runs, the CRL 5 ppm AQC check solution made from Spex sources was used as the primary control check standard. For the last two analysis runs, a 5 ppm AQC solution prepared from the Inorganic Ventures solutions was used as the primary control check standard.

Because of the separation method used to split the sample into separate portions or fractions, it was found that some samples were found to yield heterogeneous fractions, even after attempting homogenization of the fractions by grinding/mixing in a laboratory ball mill. This was evidenced from the large RPD values for laboratory duplicates while many spiked samples showed acceptable matrix spike recovery. For a few samples, then, triplicate determinations were performed and average results were reported.

Although no formal report forms were provided with the Case deliverables, the additional information from the two back up element lines (the Pb 182 nm and the Cd 214 nm line) agreed with the Pb and Cd values reported. Overall, the corresponding element values agreed within 2% to 4%.

A one day check of IDL values for the Pb and Cd lines was conducted during analysis Run 966A. A table listing these values is included with the Case deliverables. These IDL values are in line (i.e., they agree) with values obtained from earlier TJA 61 studies.